

The first phosphoramidate–mercury(II) complex with a $\text{Cl}_2\text{Hg–OP}[\text{N}(\text{C})(\text{C})]_3$ segment

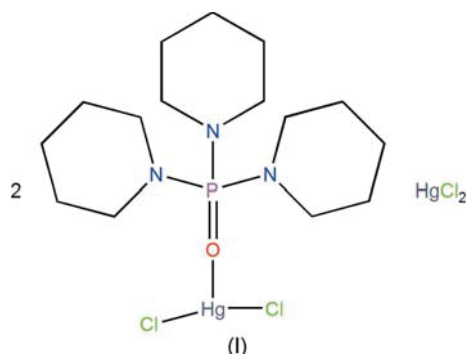
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Mercury(II) exhibits a strong preference for linear coordination which has been attributed to relativistic effects splitting the $6p$ orbitals and promoting sp hybridization. If the two ligands attached to the mercury(II) ion are weak donors, the metal ion can act as a good Lewis acid and expand its coordination number. Moreover, mercury has a special affinity for softer bases, such as S and N atoms, and has much less affinity for hard bases, such as those including an O atom. The asymmetric unit of dichlorido[tris(piperidin-1-yl)phosphane oxide- κO]mercury(II)–dichloridomercury(II) (2/1), $[\text{HgCl}_2\{(\text{C}_5\text{H}_{10}\text{N})_3\text{PO}\}]_2 \cdot [\text{HgCl}_2]$, is composed of one $\text{HgCl}_2\{(\text{C}_5\text{H}_{10}\text{N})_3\text{PO}\}$ complex and one half of a discrete HgCl_2 entity located on an inversion centre. The coordination environment around the Hg^{II} centre in the complex component is a distorted T-shape. Bond-valence-sum calculations confirm the three-coordination mode of the Hg^{II} atom of the complex molecule. The noncovalent nature of the $\text{Hg} \cdots \text{Cl}$ and $\text{Hg} \cdots \text{O}$ interactions in the structure are discussed.

1. Introduction

Mercuric coordination compounds including ligands with different donor atoms (typically, sulfur, nitrogen and oxygen) have been investigated (Lippard, 1990). The +2 oxidation state is the most common for Hg and is also the main oxidation state of mercury in nature. The coordination chemistry of mercury(II) differs from most other transition metals due to its d^{10} configuration and large size (Sahebalzamani *et al.*, 2010).



Mercury(II) exhibits a strong preference for linear coordination (Canty & Marker, 1976) which has been attributed to relativistic effects splitting the $6p$ orbitals and promoting sp hybridization (Thomas & Gaillard, 2015). It should be noted that if the two ligands attached to the mercury(II) ion are weak donors, *e.g.* chloride in HgCl_2 , the metal ion can act as a good Lewis acid and expand its coordination number (Fisher

& Drago, 1975). Moreover, mercury has a special affinity for softer bases, such as S and N atoms, and has much less affinity for hard bases, such as those including an O atom (Meyer & Nockemann, 2003).

In this paper, we describe the synthesis and structural characterization of a new three-coordinated mercury(II) complex with a tris(piperidin-1-yl)phosphane oxide *O*-donor ligand. A search of the Cambridge Structural Database (CSD, Version 5.36, with updates to November 2014; Groom & Allen, 2014) revealed that: (i) the structure of (I) is the first example of a three-coordinated Hg^{II} complex with a Cl₂Hg—O=P[N(C)(C)]₃ segment; (ii) there are only two phosphoric triamide Hg^{II} structures present, namely bis(dimesylamide- κ N)(hexamethylphosphoramidate- κ O)mercury(II) (CSD refcode JUWNOE; Blaschette *et al.*, 1993) and bis(μ -hexamethylphosphoramidate)tris(μ -perfluoro-*o*-phenylene)trimercury(II) (CAMFEC; Tikhonova *et al.*, 2002), the preparation of both of which used the commercial material [(CH₃)₂N]₃PO; (iii) the (C₅H₁₀N)₃PO ligand appears in only two complex structures, namely tris(isothiocyanato)tris(tripiperidinylphosphine oxide)-praseodymium(III) (MALMAO; da Silva *et al.*, 2005) and bis(nitrato- κ^2 O,*O'*)dioxidobis(tripiperidinylphosphine oxide- κ O)uranium (LOPVUH; de Aquino *et al.*, 2000).

2. Experimental

2.1. Synthesis and crystallization

2.1.1. Synthesis of tris(piperidin-1-yl)phosphane oxide, (C₅H₁₀N)₃PO. The preparation of tris(piperidin-1-yl)phosphane oxide was reported previously as the by-product of the synthesis of [(C₅H₁₀N)₄P]Br (Schiemenz *et al.*, 2001). The synthesis of (C₅H₁₀N)₃PO in the present case was performed in a different way. A dry acetonitrile solution of piperidine (1.2 M, 10 ml) was added dropwise to a solution of P(O)Cl₃ (2 mmol) in the same solvent (30 ml) at 273 K. After stirring for 4 h, the precipitated amine hydrochloride salt (C₅H₁₀NH·HCl) was filtered off and the acetonitrile solution of tris(piperidin-1-yl)phosphane oxide was used in the subsequent preparation of the title compound.

2.1.2. Synthesis of [HgCl₂{(C₅H₁₀N)₃PO}]₂·[HgCl₂], (I). A solution of HgCl₂ (1 mmol) in methanol (10 ml) was added dropwise to the acetonitrile solution of (C₅H₁₀N)₃PO (2 mmol, 50 ml) and the resulting solution stirred for 48 h under reflux. After cooling to room temperature, colourless block-shaped crystals of (I) were obtained within a few days.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were included in calculated positions and treated as riding atoms, with C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Attempts to model the disorder of the piperidine ring containing atom N2 were unsuccessful. Instead rigid-bond and similarity restraints were applied to atoms C6, C7, C8, C9 and C10.

Table 1
Experimental details.

Crystal data	
Chemical formula	[HgCl ₂ (C ₁₅ H ₃₀ N ₃ OP)] ₂ ·[HgCl ₂]
M_r	1413.25
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	300
a, b, c (Å)	11.1182 (15), 10.5995 (8), 19.338 (3)
β (°)	96.337 (11)
V (Å ³)	2265.0 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	10.60
Crystal size (mm)	0.23 × 0.16 × 0.10
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (<i>X-SHAPE</i> ; Stoe & Cie, 2002)
$T_{\text{min}}, T_{\text{max}}$	0.066, 0.133
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	27440, 4041, 3336
R_{int}	0.061
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.599
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.036, 0.074, 1.05
No. of reflections	4041
No. of parameters	223
No. of restraints	31
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.66, -0.79

Computer programs: *X-AREA* (Stoe & Cie, 2009), *X-RED32* (Stoe & Cie, 2009), *SUPERFLIP* (Palatinus & Chapuis, 2007), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 1999) and *ORTEP-3 for Windows* (Farrugia, 2012).

3. Results and discussion

The asymmetric unit of (I) is composed of one HgCl₂-{(C₅H₁₀N)₃PO} complex and one half of a HgCl₂ component (Fig. 1). In the discrete HgCl₂ component, the Hg2 atom is located on an inversion centre and hence shows an ideal linear coordination environment.

The Hg1—Cl bond lengths in the mercury(II)–phosphoric triamide complex of (I) are consistent with those reported for other complexes of Hg^{II} chloride (Li *et al.*, 2012; Kubo *et al.*, 2000) and are longer than the two equal Hg—Cl bond lengths in the discrete HgCl₂ entity (Table 2). The Hg1—O1 bond length is comparable to similar bonds in analogous structures (Yang *et al.*, 2010).

In the HgCl₂{(C₅H₁₀N)₃PO} complex, Cl1—Hg1—Cl2 is distorted by about 16° from a linear bonding arrangement due to the presence of the coordinated phosphoric triamide molecule. The coordinated O atom forms Cl1—Hg1—O1 and Cl2—Hg1—O1 angles that deviate from 90° towards higher angles, by about 16° and less than 1°, respectively (Table 2).

Khavasi & Azhdari Tehrani (2013) introduced the simple equation $\tau_3 = |[(\alpha + \beta + \gamma)/360] - |(\alpha - 120)/60||$ for the calculation of geometry index τ_3 for three-coordinated compounds. In this equation, α is the largest angle in the three-coordinated compound and the values of τ_3 range from 1.00 for a perfect trigonal planar geometry to zero for a perfect T-shaped geometry. The τ_3 index for the three-coordinated

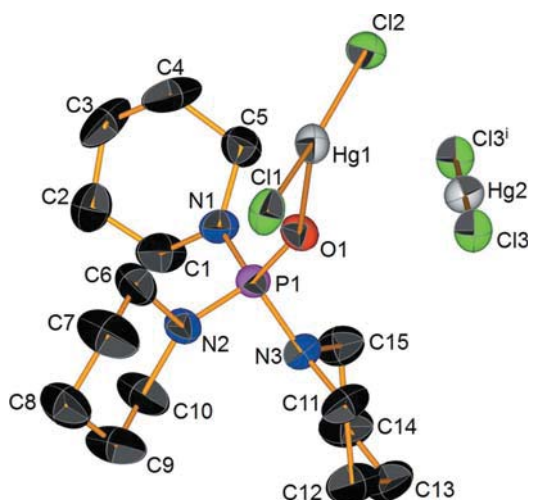


Figure 1

Displacement ellipsoid plot (30% probability level) for the $[\text{HgCl}_2 \cdot ((\text{C}_5\text{H}_{10}\text{N})_3\text{PO})]$ and HgCl_2 components in (I), showing the atom-labelling scheme. H atoms have been omitted for clarity. [Symmetry code: (i) $-x, -y + 2, -z + 2$.]

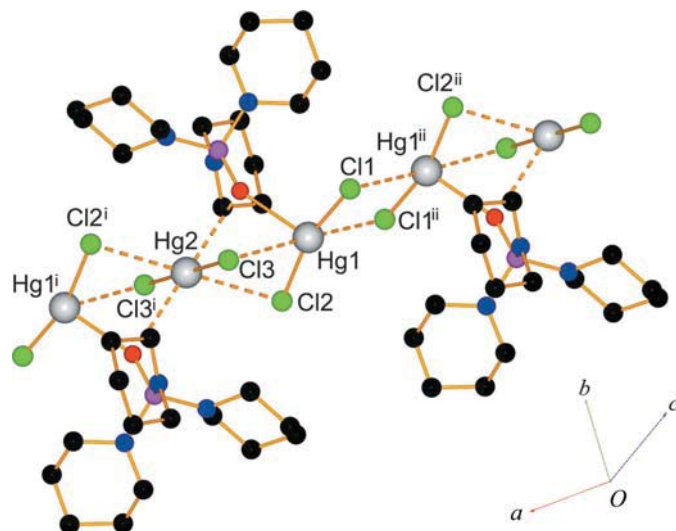


Figure 2

A view of linear arrangement of components parallel to $[100]$ built from $\text{Hg} \cdots \text{Cl}$ and $\text{Hg} \cdots \text{O}$ interactions (dashed lines). H atoms have been omitted for clarity. [Symmetry codes: (i) $-x, -y + 2, -z + 2$; (ii) $-x + 1, -y + 2, -z + 2$.]

Hg1 complex of (I) is 0.27, which implies a distorted T-shape geometry (Tiekink, 1987).

Using a van der Waals radius of 1.70 Å for mercury, 1.75 Å for chlorine and 1.50 Å for oxygen (Batsanov, 2001), four different noncovalent interactions are considered in (I), namely $\text{Hg1} \cdots \text{Cl3}$ [3.154 (2) Å], $\text{Hg2} \cdots \text{Cl2}$ [3.166 (2) Å], $\text{Hg2} \cdots \text{O1}$ [2.940 (5) Å] and $\text{Hg1} \cdots \text{Cl1}^{\text{ii}}$ [3.121 (2) Å; symmetry code: (ii) $-x + 1, -y + 2, -z + 2$]. The two last interactions take part in forming a chain along the a direction (Fig. 2) and the other two help to stabilize the aggregation with no effect on the pattern of the extended structure formed. Such simultaneously close and distant bonded atoms are usual for mercury(II) (Canty, 1980). A view of the linear chains in the crystal structure of (I) is shown in Fig. 3.

In order to prove the noncovalent nature of the $\text{Hg} \cdots \text{Cl}$ interaction involving atom Hg1 in the three-coordinate complex, bond-valence-sum calculations were performed using the equation $S_{ij} = \exp[(R_0 - R_{ij})/b_{ij}]$, where S_{ij} is the bond valence portion between atoms i and j , R_0 is the standard value of the bond distance for atoms i and j , R_{ij} is the actual bond distance between atoms i and j , and b_{ij} is a constant (Brown & Altermatt, 1985). Such a calculation for the $\text{Hg}[\text{Cl}]_2[\text{O}]$ segment provides a bond-valence-sum (BVS)

Table 2
Selected geometric parameters (Å, °).

$\text{Hg1}-\text{Cl1}$	2.301 (2)	$\text{P1}-\text{O1}$	1.497 (4)
$\text{Hg1}-\text{Cl2}$	2.303 (2)	$\text{P1}-\text{N1}$	1.625 (6)
$\text{Hg1}-\text{O1}$	2.474 (4)	$\text{P1}-\text{N3}$	1.634 (6)
$\text{Hg2}-\text{Cl3}$	2.2829 (17)	$\text{P1}-\text{N2}$	1.636 (6)
$\text{Cl1}-\text{Hg1}-\text{Cl2}$	163.79 (8)	$\text{P1}-\text{O1}-\text{Hg1}$	152.6 (3)
$\text{Cl1}-\text{Hg1}-\text{O1}$	105.57 (13)	$\text{O1}-\text{P1}-\text{N1}$	109.4 (3)
$\text{Cl2}-\text{Hg1}-\text{O1}$	90.58 (12)	$\text{O1}-\text{P1}-\text{N2}$	111.2 (3)
$\text{Cl3}^{\text{i}}-\text{Hg2}-\text{Cl3}$	180.0	$\text{O1}-\text{P1}-\text{N3}$	114.2 (3)

Symmetry code: (i) $-x, -y + 2, -z + 2$.

value of 2.142 valence units (v.u.) for Hg1 , considering two $\text{Hg}-\text{Cl}$ and one $\text{Hg}-\text{O}$ bonds (with R_{ij} as given in Table 2 for the $\text{Hg1}-\text{Cl1}$, $\text{Hg1}-\text{Cl2}$ and $\text{Hg1}-\text{O1}$ bond lengths, $R_0 = 2.28$ Å for $\text{Hg}-\text{Cl}$ and 1.972 Å for $\text{Hg}-\text{O}$, and $b_{ij} = 0.37$; Brown, <http://www.iucr.org/resources/data/datasets/bond-valence-parameters>). Considering the $\text{Hg1} \cdots \text{Cl1}^{\text{ii}}$ and $\text{Hg1} \cdots \text{Cl3}$ interactions, the BVSs are calculated as 2.245 and 2.339 v.u., respectively, confirming the noncovalent nature of these interactions.

The P atom in the phosphoric triamide ligand adopts a slightly distorted $\text{P}[\text{O}][\text{N}]_3$ tetrahedral environment and the

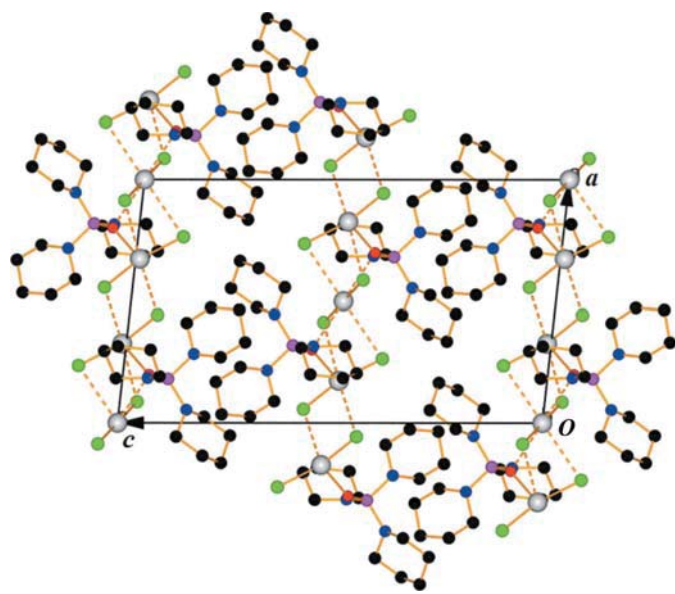


Figure 3

A view of the crystal packing of (I), viewed along the b axis, showing adjacent linear chains. The $\text{Hg} \cdots \text{Cl}$ and $\text{Hg} \cdots \text{O}$ interactions are shown as dashed lines and H atoms have been omitted for clarity.

Hg1–O1–P1 unit is significantly bent, with an angle of 152.6 (3)°. Moreover, the P1=O1 bond length in complex (I) is longer than the P=O double-bond length in phosphoric triamide compounds, for example, longer by about 0.008 Å compared with tris(morpholino)phosphine oxide (BIVYAG; Romming & Songstad, 1982), which is the closest structure to the tris(piperidin-1-yl)phosphane oxide discussed here.

The six-membered piperidine rings in the phosphoric triamide ligand adopt near-chair conformations.

Acknowledgements

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References

- Aquino, A. R. de, Bombieri, G., Isolani, P. C., Vicentini, G. & Zukerman-Schpector, J. (2000). *Inorg. Chim. Acta*, **306**, 102–106.
- Batsanov, S. S. (2001). *Inorg. Mater.* **37**, 871–885.
- Blaschette, A., Jones, P. G., Michalides, A. & Linoh, K. (1993). *Z. Anorg. Allg. Chem.* **619**, 392–402.
- Brandenburg, K. & Putz, H. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Brown, I. D. & Altermatt, D. (1985). *Acta Cryst.* **B41**, 244–247.
- Canty, A. J. (1980). *Inorg. Chim. Acta*, **45**, L225–L227.
- Canty, A. J. & Marker, A. (1976). *Inorg. Chem.* **15**, 425–430.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Fisher, K. J. & Drago, R. S. (1975). *Inorg. Chem.* **14**, 2804–2808.
- Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
- Khavasi, H. R. & Azhdari Tehrani, A. (2013). *Inorg. Chem.* **52**, 2891–2905.
- Kubo, K., Kato, N., Mori, A. & Takeshita, H. (2000). *Acta Cryst.* **C56**, 644–646.
- Li, Y., Liu, Q.-K., Ma, J.-P. & Dong, Y.-B. (2012). *Acta Cryst.* **C68**, m152–m155.
- Lippard, S. J. (1990). Editor. *Progress in Inorganic Chemistry: Bioinorganic Chemistry*, Vol. 38. London: John Wiley & Sons.
- Meyer, G. & Nockemann, P. (2003). *Z. Anorg. Allg. Chem.* **629**, 1447–1461.
- Palatinus, L. & Chapuis, G. (2007). *J. Appl. Cryst.* **40**, 786–790.
- Romming, C. & Songstad, J. (1982). *Acta Chem. Scand. Ser. A*, **36**, 665–671.
- Sahebalzamani, H., Ghammamy, S., Mehrani, K. & Salimi, F. (2010). *Der Chem. Sin.* **1**, 39–44.
- Schiemenz, B., Wessel, T., Pfirmann, R. & Beck, A. (2001). Eur Patent EP 1 070 724 A1.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Silva, M. F. P. da, Zukerman-Schpector, J., Vicentini, G. & Isolani, P. C. (2005). *Inorg. Chim. Acta*, **358**, 796–800.
- Stoe & Cie (2002). *X-AREA*. Stoe & Cie GmbH, Darmstadt, Germany.
- Stoe & Cie (2009). *X-RED32*. Stoe & Cie GmbH, Darmstadt, Germany.
- Thomas, S. A. & Gaillard, J. F. (2015). *J. Phys. Chem. A*, **119**, 2878–2884.
- Tiekink, E. R. T. (1987). *Acta Cryst.* **C43**, 448–450.
- Tikhonova, I. A., Dolgushin, F. M., Tugashov, K. I., Petrovskii, P. V., Furin, G. G. & Shur, V. B. (2002). *J. Organomet. Chem.* **654**, 123–131.
- Yang, X. J., Liu, X., Liu, Y., Hao, Y. & Wu, B. (2010). *Polyhedron*, **29**, 934–940.

supporting information

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The first phosphoramidate–mercury(II) complex with a Cl₂Hg–OP[N(C)(C)]₃ segment

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2009); cell refinement: *X-AREA* (Stoe & Cie, 2009); data reduction: *X-RED32* (Stoe & Cie, 2009); program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg & Putz, 1999) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

Dichlorido[tris(piperidin-1-yl)phosphane oxide-κO]mercury(II)–dichloridomercury(II) (2/1)

Crystal data

[HgCl₂(C₁₅H₃₀N₃OP)][HgCl₂]
M_r = 1413.25
Monoclinic, *P*2₁/*n*
a = 11.1182 (15) Å
b = 10.5995 (8) Å
c = 19.338 (3) Å
β = 96.337 (11)°
V = 2265.0 (5) Å³
Z = 2

F(000) = 1340
D_x = 2.072 Mg m⁻³
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 19807 reflections
θ = 1.1–25.6°
μ = 10.60 mm⁻¹
T = 300 K
Block, less
0.23 × 0.16 × 0.10 mm

Data collection

Stoe IPDS 2
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 6.67 pixels mm⁻¹
rotation method scans
Absorption correction: integration
(*X-SHAPE*; Stoe & Cie, 2002)
T_{min} = 0.066, *T_{max}* = 0.133

27440 measured reflections
4041 independent reflections
3336 reflections with *I* > 2σ(*I*)
R_{int} = 0.061
θ_{max} = 25.2°, *θ_{min}* = 2.0°
h = -13→13
k = -12→12
l = -23→23

Refinement

Refinement on *F*²
Least-squares matrix: full
R[*F*² > 2σ(*F*²)] = 0.036
wR(*F*²) = 0.074
S = 1.05
4041 reflections
223 parameters
31 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
w = 1/[σ²(*F_o*²) + (0.0217*P*)² + 5.1731*P*]
where *P* = (*F_o*² + 2*F_c*²)/3
(Δ/σ)_{max} = 0.002
Δρ_{max} = 0.66 e Å⁻³
Δρ_{min} = -0.79 e Å⁻³

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. DELU and SIME instructions in *SHELXL97* (Sheldrick, 2008) were applied to C6, C7, C8, C9, and C10 in order to limit the disorder in the corresponding ring. A splitting of the ring in two different parts was not solving the problems of displacement of some atoms in the ring. With these two instructions, the problem is not solved but limited.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1911 (11)	0.5083 (7)	0.9128 (5)	0.114 (4)
H1A	0.1217	0.4599	0.9239	0.137*
H1B	0.1886	0.5125	0.8626	0.137*
C2	0.3005 (10)	0.4461 (10)	0.9412 (6)	0.114 (3)
H2A	0.3693	0.4895	0.9255	0.137*
H2B	0.3003	0.3604	0.9235	0.137*
C3	0.2957 (11)	0.5713 (9)	1.0499 (5)	0.128 (4)
H3A	0.2892	0.5622	1.0992	0.153*
H3B	0.3652	0.6243	1.0445	0.153*
C4	0.3152 (10)	0.4423 (9)	1.0188 (6)	0.129 (4)
H4A	0.3958	0.4126	1.0352	0.155*
H4B	0.2575	0.3831	1.0346	0.155*
C5	0.1835 (8)	0.6340 (8)	1.0157 (4)	0.088 (2)
H5A	0.1126	0.5889	1.0274	0.105*
H5B	0.1792	0.7198	1.0326	0.105*
C6	0.4104 (8)	0.7807 (10)	0.8631 (5)	0.101 (3)
H6A	0.4135	0.8382	0.9023	0.121*
H6B	0.4504	0.7031	0.8792	0.121*
C7	0.4737 (11)	0.8352 (14)	0.8102 (6)	0.151 (5)
H7A	0.5585	0.8434	0.8280	0.181*
H7B	0.4424	0.9194	0.8003	0.181*
C8	0.4647 (10)	0.7608 (12)	0.7427 (5)	0.123 (3)
H8A	0.5102	0.6828	0.7495	0.148*
H8B	0.4980	0.8096	0.7069	0.148*
C9	0.3312 (10)	0.7319 (12)	0.7207 (5)	0.131 (4)
H9A	0.2905	0.8094	0.7049	0.158*
H9B	0.3253	0.6739	0.6816	0.158*
C10	0.2703 (10)	0.6786 (12)	0.7747 (5)	0.125 (4)
H10A	0.3019	0.5947	0.7851	0.150*
H10B	0.1849	0.6705	0.7584	0.150*
C11	0.0250 (9)	0.8724 (8)	0.7978 (5)	0.100 (3)
H11A	0.0994	0.9109	0.7864	0.120*
H11B	-0.0172	0.9336	0.8237	0.120*
C12	-0.0526 (11)	0.8387 (12)	0.7320 (5)	0.130 (4)
H12A	-0.0736	0.9149	0.7057	0.156*
H12B	-0.0069	0.7847	0.7039	0.156*

C13	-0.1650 (10)	0.7727 (11)	0.7461 (5)	0.122 (4)
H13A	-0.2095	0.7465	0.7024	0.147*
H13B	-0.2159	0.8301	0.7690	0.147*
C14	-0.1355 (9)	0.6590 (11)	0.7915 (5)	0.117 (3)
H14A	-0.0926	0.5973	0.7665	0.141*
H14B	-0.2098	0.6205	0.8032	0.141*
C15	-0.0569 (8)	0.6978 (9)	0.8585 (5)	0.098 (3)
H15A	-0.1020	0.7549	0.8851	0.118*
H15B	-0.0361	0.6237	0.8867	0.118*
C11	0.44342 (18)	1.12472 (19)	0.93393 (12)	0.0866 (6)
C12	0.2359 (2)	0.9726 (2)	1.10663 (10)	0.0895 (6)
C13	0.08680 (18)	1.17631 (17)	0.95927 (11)	0.0783 (5)
Hg1	0.32657 (3)	1.02803 (3)	1.00954 (2)	0.07212 (11)
Hg2	0.0000	1.0000	1.0000	0.07027 (13)
O1	0.1987 (4)	0.8749 (4)	0.9383 (2)	0.0708 (12)
P1	0.18104 (17)	0.76084 (17)	0.89264 (9)	0.0635 (5)
N1	0.1848 (6)	0.6350 (5)	0.9408 (3)	0.0774 (17)
N2	0.2835 (5)	0.7536 (6)	0.8382 (3)	0.0714 (15)
N3	0.0539 (5)	0.7600 (5)	0.8414 (3)	0.0689 (15)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.192 (12)	0.061 (5)	0.084 (6)	0.014 (6)	-0.010 (7)	-0.005 (4)
C2	0.123 (8)	0.091 (6)	0.128 (9)	0.028 (6)	0.009 (7)	0.006 (6)
C3	0.180 (11)	0.096 (7)	0.089 (6)	-0.010 (7)	-0.060 (7)	0.012 (5)
C4	0.127 (8)	0.077 (6)	0.165 (11)	0.016 (6)	-0.065 (7)	0.016 (6)
C5	0.109 (7)	0.075 (5)	0.082 (6)	0.008 (5)	0.019 (5)	0.002 (4)
C6	0.086 (6)	0.127 (8)	0.092 (6)	-0.005 (5)	0.017 (5)	-0.036 (5)
C7	0.122 (8)	0.221 (13)	0.114 (8)	-0.076 (8)	0.033 (6)	-0.047 (7)
C8	0.113 (6)	0.164 (10)	0.099 (6)	-0.024 (7)	0.039 (6)	-0.021 (6)
C9	0.133 (8)	0.192 (11)	0.070 (6)	-0.052 (8)	0.014 (5)	-0.012 (6)
C10	0.115 (7)	0.178 (10)	0.086 (6)	-0.032 (7)	0.030 (6)	-0.043 (6)
C11	0.106 (7)	0.087 (6)	0.100 (7)	0.003 (5)	-0.023 (5)	0.022 (5)
C12	0.137 (10)	0.147 (10)	0.099 (8)	0.007 (8)	-0.020 (7)	0.014 (7)
C13	0.110 (9)	0.141 (10)	0.105 (8)	0.020 (7)	-0.037 (6)	-0.013 (7)
C14	0.105 (8)	0.137 (9)	0.103 (7)	-0.031 (7)	-0.021 (6)	-0.015 (6)
C15	0.085 (6)	0.115 (7)	0.091 (6)	-0.017 (5)	-0.003 (5)	0.010 (5)
C11	0.0726 (12)	0.0741 (12)	0.1148 (16)	0.0062 (10)	0.0178 (11)	0.0170 (11)
C12	0.0850 (13)	0.1108 (16)	0.0726 (12)	0.0067 (12)	0.0082 (10)	-0.0037 (11)
C13	0.0775 (12)	0.0641 (10)	0.0962 (14)	-0.0030 (9)	0.0231 (10)	0.0111 (9)
Hg1	0.06359 (18)	0.06830 (18)	0.0848 (2)	0.00149 (14)	0.00990 (14)	-0.00687 (14)
Hg2	0.0711 (3)	0.0643 (2)	0.0750 (3)	-0.01132 (18)	0.0064 (2)	0.00707 (18)
O1	0.083 (3)	0.058 (3)	0.071 (3)	-0.006 (2)	0.006 (2)	-0.014 (2)
P1	0.0709 (12)	0.0579 (10)	0.0600 (11)	0.0033 (9)	-0.0001 (9)	-0.0061 (8)
N1	0.113 (5)	0.052 (3)	0.063 (4)	0.013 (3)	-0.008 (3)	-0.002 (3)
N2	0.069 (4)	0.077 (4)	0.067 (4)	-0.004 (3)	0.004 (3)	-0.016 (3)
N3	0.065 (4)	0.068 (4)	0.071 (4)	-0.001 (3)	-0.001 (3)	0.006 (3)

Geometric parameters (Å, °)

C1—C2	1.438 (13)	C10—N2	1.457 (10)
C1—N1	1.452 (9)	C10—H10A	0.9700
C1—H1A	0.9700	C10—H10B	0.9700
C1—H1B	0.9700	C11—N3	1.475 (9)
C2—C4	1.493 (13)	C11—C12	1.500 (12)
C2—H2A	0.9700	C11—H11A	0.9700
C2—H2B	0.9700	C11—H11B	0.9700
C3—C5	1.501 (12)	C12—C13	1.483 (15)
C3—C4	1.518 (14)	C12—H12A	0.9700
C3—H3A	0.9700	C12—H12B	0.9700
C3—H3B	0.9700	C13—C14	1.507 (14)
C4—H4A	0.9700	C13—H13A	0.9700
C4—H4B	0.9700	C13—H13B	0.9700
C5—N1	1.450 (9)	C14—C15	1.536 (11)
C5—H5A	0.9700	C14—H14A	0.9700
C5—H5B	0.9700	C14—H14B	0.9700
C6—C7	1.426 (13)	C15—N3	1.467 (10)
C6—N2	1.469 (10)	C15—H15A	0.9700
C6—H6A	0.9700	C15—H15B	0.9700
C6—H6B	0.9700	Hg1—Cl1	2.301 (2)
C7—C8	1.519 (13)	Hg1—Cl2	2.303 (2)
C7—H7A	0.9700	Hg1—O1	2.474 (4)
C7—H7B	0.9700	Hg2—Cl3	2.2829 (17)
C8—C9	1.530 (14)	Hg2—Cl3 ⁱ	2.2829 (18)
C8—H8A	0.9700	P1—O1	1.497 (4)
C8—H8B	0.9700	P1—N1	1.625 (6)
C9—C10	1.423 (13)	P1—N3	1.634 (6)
C9—H9A	0.9700	P1—N2	1.636 (6)
C9—H9B	0.9700		
C2—C1—N1	110.8 (8)	C9—C10—H10A	109.0
C2—C1—H1A	109.5	N2—C10—H10A	109.0
N1—C1—H1A	109.5	C9—C10—H10B	109.0
C2—C1—H1B	109.5	N2—C10—H10B	109.0
N1—C1—H1B	109.5	H10A—C10—H10B	107.8
H1A—C1—H1B	108.1	N3—C11—C12	111.2 (8)
C1—C2—C4	113.0 (9)	N3—C11—H11A	109.4
C1—C2—H2A	109.0	C12—C11—H11A	109.4
C4—C2—H2A	109.0	N3—C11—H11B	109.4
C1—C2—H2B	109.0	C12—C11—H11B	109.4
C4—C2—H2B	109.0	H11A—C11—H11B	108.0
H2A—C2—H2B	107.8	C13—C12—C11	112.0 (9)
C5—C3—C4	111.9 (7)	C13—C12—H12A	109.2
C5—C3—H3A	109.2	C11—C12—H12A	109.2
C4—C3—H3A	109.2	C13—C12—H12B	109.2
C5—C3—H3B	109.2	C11—C12—H12B	109.2

C4—C3—H3B	109.2	H12A—C12—H12B	107.9
H3A—C3—H3B	107.9	C12—C13—C14	110.5 (9)
C2—C4—C3	111.6 (8)	C12—C13—H13A	109.6
C2—C4—H4A	109.3	C14—C13—H13A	109.6
C3—C4—H4A	109.3	C12—C13—H13B	109.6
C2—C4—H4B	109.3	C14—C13—H13B	109.6
C3—C4—H4B	109.3	H13A—C13—H13B	108.1
H4A—C4—H4B	108.0	C13—C14—C15	110.1 (9)
N1—C5—C3	110.1 (7)	C13—C14—H14A	109.6
N1—C5—H5A	109.6	C15—C14—H14A	109.6
C3—C5—H5A	109.6	C13—C14—H14B	109.6
N1—C5—H5B	109.6	C15—C14—H14B	109.6
C3—C5—H5B	109.6	H14A—C14—H14B	108.1
H5A—C5—H5B	108.2	N3—C15—C14	110.2 (7)
C7—C6—N2	112.1 (8)	N3—C15—H15A	109.6
C7—C6—H6A	109.2	C14—C15—H15A	109.6
N2—C6—H6A	109.2	N3—C15—H15B	109.6
C7—C6—H6B	109.2	C14—C15—H15B	109.6
N2—C6—H6B	109.2	H15A—C15—H15B	108.1
H6A—C6—H6B	107.9	C11—Hg1—Cl2	163.79 (8)
C6—C7—C8	114.5 (10)	C11—Hg1—O1	105.57 (13)
C6—C7—H7A	108.6	C12—Hg1—O1	90.58 (12)
C8—C7—H7A	108.6	C13 ⁱ —Hg2—Cl3	180.0
C6—C7—H7B	108.6	P1—O1—Hg1	152.6 (3)
C8—C7—H7B	108.6	O1—P1—N1	109.4 (3)
H7A—C7—H7B	107.6	N1—P1—N3	108.0 (3)
C7—C8—C9	108.2 (9)	O1—P1—N2	111.2 (3)
C7—C8—H8A	110.0	O1—P1—N3	114.2 (3)
C9—C8—H8A	110.0	N1—P1—N2	110.8 (3)
C7—C8—H8B	110.0	N3—P1—N2	103.1 (3)
C9—C8—H8B	110.0	C5—N1—C1	111.8 (6)
H8A—C8—H8B	108.4	C5—N1—P1	125.2 (5)
C10—C9—C8	113.6 (9)	C1—N1—P1	123.0 (5)
C10—C9—H9A	108.8	C10—N2—C6	112.8 (7)
C8—C9—H9A	108.8	C10—N2—P1	123.7 (6)
C10—C9—H9B	108.8	C6—N2—P1	119.5 (5)
C8—C9—H9B	108.8	C15—N3—C11	110.8 (6)
H9A—C9—H9B	107.7	C15—N3—P1	123.9 (5)
C9—C10—N2	113.0 (9)	C11—N3—P1	117.5 (5)
N1—C1—C2—C4	55.8 (13)	N3—P1—N1—C1	66.0 (9)
C1—C2—C4—C3	-49.4 (14)	N2—P1—N1—C1	-46.3 (9)
C5—C3—C4—C2	47.2 (14)	C9—C10—N2—C6	-53.4 (13)
C4—C3—C5—N1	-52.2 (11)	C9—C10—N2—P1	149.3 (8)
N2—C6—C7—C8	-52.8 (14)	C7—C6—N2—C10	52.7 (12)
C6—C7—C8—C9	50.3 (15)	C7—C6—N2—P1	-149.0 (8)
C7—C8—C9—C10	-50.1 (15)	O1—P1—N2—C10	-160.6 (8)
C8—C9—C10—N2	53.2 (15)	N1—P1—N2—C10	77.5 (8)

N3—C11—C12—C13	55.9 (12)	N3—P1—N2—C10	-37.8 (8)
C11—C12—C13—C14	-54.6 (13)	O1—P1—N2—C6	43.6 (7)
C12—C13—C14—C15	55.0 (13)	N1—P1—N2—C6	-78.3 (7)
C13—C14—C15—N3	-57.4 (12)	N3—P1—N2—C6	166.4 (6)
Hg1—O1—P1—N1	83.1 (7)	C14—C15—N3—C11	58.7 (10)
Hg1—O1—P1—N3	-155.7 (6)	C14—C15—N3—P1	-152.9 (7)
Hg1—O1—P1—N2	-39.5 (7)	C12—C11—N3—C15	-57.9 (11)
C3—C5—N1—C1	59.3 (10)	C12—C11—N3—P1	151.5 (7)
C3—C5—N1—P1	-120.2 (7)	O1—P1—N3—C15	-94.5 (7)
C2—C1—N1—C5	-61.4 (11)	N1—P1—N3—C15	27.4 (7)
C2—C1—N1—P1	118.1 (8)	N2—P1—N3—C15	144.6 (7)
O1—P1—N1—C5	10.2 (8)	O1—P1—N3—C11	51.9 (7)
N3—P1—N1—C5	-114.6 (7)	N1—P1—N3—C11	173.8 (6)
N2—P1—N1—C5	133.1 (7)	N2—P1—N3—C11	-68.9 (7)
O1—P1—N1—C1	-169.2 (8)		

Symmetry code: (i) $-x, -y+2, -z+2$.